

Standard Operating Procedure

Solids

Total Solids (TS)
Total Suspended Solids (TSS)
Total Volatile Suspended Solids (TVSS)
Total Volatile Solids (TVS)
Total Dissolved Solids (TDS)
Moisture (%H₂O)

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Authorized By: Courtney Diduck, Analytical Services Branch Head

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[N:\Environmental Standards\Analytical Services\SOP \(PDFs\)](N:\Environmental Standards\Analytical Services\SOP (PDFs))

Hyperlinks to external documents and within this document are highlighted and underlined in green font. These are functional in the electronic version of this document, when the user has permission and the document is viewed within the City of Winnipeg's intranet site from the above mentioned folder. All documents pertaining to this SOP are maintained in subfolders of the following Master Folder;

[N:\Environmental Standards\Methods\Solids \(SOP 12\)](N:\Environmental Standards\Methods\Solids (SOP 12))

Latest revisions are highlighted in blue print.

All hand-written amendments (except for spelling or grammatical errors) must be approved immediately by the Analytical Services Branch Head or Quality Assurance Officer, who shall initial and date the amendments. The procedure shall be formally revised and reissued as soon as practicable.

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All forms, reagent logs are located in;

[N:\Environmental Standards\Methods\Solids \(SOP 12\)\FORMS\SolidsForms.xlsx](N:\Environmental Standards\Methods\Solids (SOP 12)\FORMS\SolidsForms.xlsx)

Control Charts: <N:\Environmental Standards\Analytical Services \Charts\ChartGenerator.xls>

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Introduction and Scope

The measurement of solids content of water and wastewater is important for regulatory purposes and environmental protection. The solids content in the different wastewater streams is a measure of the wastewater treatment plant efficiency, and solids loading. The solids content is also used to determine the wastewater surcharge for industrial and septic waste generators.

Referenced Method

This method is modified from Standard Methods for the Examination of Water and Wastewater, 2540 Solids, approved by Standard Methods Committee 1997, Editorial Revisions 2011, with the following modifications;

- Samples are not weighed to constant weight. Instead, a much longer drying time is used for TDS & TS and a reweighed sample is included in each batch. If the reweigh is outside the established quality control limits, then the batch is dried for an additional 30 minutes and desiccated again. Sludge and samples with high solids or grease content are dried overnight (>12 hrs).
- For Total Dissolved Solids, some samples (such as Industrial wastes and leachates) cannot be filtered due to their extremely high content of particulate matter that immediately clog the filter. For these samples only, the sample is centrifuged at 4000 rpm for 20 minutes and the centrate is used for TDS.

Scope

	TSS	TVSS	TDS	TS	%TVS
Applicable Range for routine water, wastewater, and sludge	3 to 20,000 mg/L	3 to 20,000 mg/L	3 to 20,000 mg/L	3 to 20,000 mg/L 0.03 to 40%	0.03 to 100%
Minimum Detectable Limits	3 mg/L	3 mg/L	3 mg/L	3 mg/L	3 mg/L
Accuracy					
Bias (relative)	-0.017	-0.017	-0.002	0.003	0.003
Precision	0.039	0.043	0.025	0.055	0.01
Expanded Uncertainty, U_e	0.084	0.087	0.065	0.062	0.029

*percentage range applies to sludge and biosolids analysis.

Fit For Use

This method is fit for its intended use for the determination of:

- Total Suspended Solids, Total Volatile Suspended Solids, and Total Dissolved Solids in the range of 3 mg/L to 20,000 mg/L in water and wastewater;
- Total Volatile Solids in the range of 0.03% to 100% in sludge and biosolids.
- Total Solids, in the range of 3 mg/L to 20,000 mg/L in water and wastewater; and the range of 0.03% to 40% in sludge and biosolids.

Method Validation By G. Levesque, QA Officer, Date: October 23, 2009.

Last validation update by G. Levesque, QA Officer, Date: October 31, 2013.

Summary of Method

Total suspended solids are determined by filtering a well-mixed measured volume of sample through a weighed standard glass fibre filter in a gooch crucible. The residue retained on the filter is dried at $104^{\circ}\text{C} \pm 1$ to a constant weight. The increase in weight of the filter and gooch represents the total suspended solids. The filtrate from the total suspended solids can be used to determine the Total Dissolved Solids.

Volatile suspended solids are determined by igniting the residue from the total suspended solids analysis in a muffle oven at 550°C .

Total solids are determined by adding a known volume of a well-mixed sample into a weighed crucible, and after heating to dryness is dried to a constant weight. Moisture is the loss of weight due to evaporation when dried.

Volatile Solids is the weight loss after ignition of the total solids residue at 550°C .

When total solids and total suspended solids are determined on the same sample, total dissolved solids can be determined by calculation.

LIMS Test Codes

TSSwv	Total Suspended Solids weight per volume (mg/L)
TVSSwv	Total Volatile Suspended Solids weight per volume (mg/L)
TVSS%	Total Volatile Suspended Solids %
TSwv	Total Solids weight per volume (mg/L)
TS%	Total Solids %
TVS%	Total Volatile Solids %
TDSwv	Total Dissolved Solids weight per volume (mg/L)
%Moisture	Moisture, the amount of water in the sample (calculated from Total Solids)

Sample Requirements

Sample History

Some samples are collected by internal clients and submitted/delivered directly to the laboratory. Routine samples are collected by laboratory personnel assigned to the task following SOP#17, Sampling.

Field or Laboratory Filtration

Not required

Preservation

If the test is not performed **within 2 hours of sample reception at the laboratory**, the sample must be stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

Holding Time

Analysis is normally performed within 24 hours of sample collection. Refrigerated samples can be held for no longer than seven (7) days.

Sample Pre-treatment

Large non-homogenous particles should be excluded from the test aliquot if it is determined that their inclusion is not desired in the final results. Floating oil and grease, if present, should be included in the sample and dispersed by vigorous stirring 5 times to create a vortex before withdrawing a sub-sample for filtration.

Floating debris (excluding oil & grease) is excluded.

Allow samples to warm to room temperature before analysis.

Containers

Samples should be collected in a plastic or glass bottle and transported to the laboratory as soon as possible for analysis.

Volume Required

Excessive residue on the filter can produce a water trapping crust, which can result in sample loss, or incomplete drying. The sample size should be limited to produce a residue of less than 200 mg. Samples containing visible suspended solids require less volume for analysis. For water samples containing little or no visible suspended solids a greater sample volume may be required for analysis. Collect a minimum of 500 mL for turbid samples and 1 litre for water samples.

Interferences

Samples with high dissolved solids may cause positive interferences. These effects can be minimized with adequate washing to remove dissolved solids, which may get trapped in the filter pores.

Filter clogging, from excessive solids captured in the filter, may prolong filtration time and produce high results. To overcome this, use a smaller sample volume for filtration, or use a larger size filter.

Chemicals & Reagents

Reagent Preparation Logs

Document the preparation of all reagents; calibration stock solutions, reference material stock solutions, and working standards on the reagent preparation log sheets behind Tab 3. Label reagents with; Material Name, Date Prepared, and Reagent Prep Log Reference Number or LIMS ID #. All Reagent preparation forms are available in [SolidsForms.xlsx](#). All Certified Reference Materials are entered into the LIMS, Chemicals and Standards Module and a copy of in the COA is scanned (downloaded) into the COA folder.

De-ionized/R.O. Water; is used when making any reagent unless otherwise stated.

Expiry Dates

Unless specified, all reagents in solid form have an expiry date of 5 years when unopened and 3 years from the date opened. Purchased reagents in liquid form have an expiry date of 3 years unopened and 12 months opened. All prepared reagents are considered "opened" on the date made and therefore never exceed 12 months. If the vendors' expiry date is less than these default dates, then use the vendors, i.e., always use the shortest date.

Diatomaceous Earth Pre-preparation (for HRM)

The diatomaceous earth is prepared in the following manner to remove the finest particles which may not get trapped in the glass filter paper.

1. Prepare the Diatomaceous Earth by placing approximately 50 grams into a 2-liter beaker.
2. Add 2 litres of deionised water, stirring vigorously for 5 minutes.
3. Allow settling for 30 minutes.
4. Decant all of the liquid. Transfer the solid into a smaller beaker and dry overnight at $104 \pm 1^{\circ}\text{C}$.
5. Remove from oven, cool for 15-20 minutes at room temperature, then transfer into desiccators until room temperature is reached. Transfer to a clean plastic wide-mouth bottle and label. Store in the desiccator. **Diatomaceous Earth** does not expire.
6. Pre-weigh several standards ahead of time into separate 25 mL vials. Label, cap and store in a desiccator.

TSS HRM, 100 mg/L (& 1,000 mg/L)

Enter the standard in the Chemical & Standard module of the LIMS as a Reference Material in order to use it in the QC Module. (Appears in drop-down list.)

Larger quantities can be prepared by multiplying the weight of the solid in the same volume of water;

1. Accurately weigh 0.204* grams into a clean oven dried glass vial with screw cap. Record the weight.
2. Put 1.5 L of deionised water into a 2-liter volumetric flask.
3. Open the bottle carefully and transfer the contents into the volumetric flask using a funnel.
4. Ensure that the entire standard is added to the flask by carefully rinsing the bottle and the cap three to four times with deionised water. Add wash into the volumetric flask.
5. Dilute up to 2000-ml mark with deionised water and mix thoroughly.
6. Mix thoroughly again before transferring into a 2 L plastic container and label with expiry date of 6 months.

To prepare a 1,000 mg/L, weight ten times (10 x) the amount of Diatomaceous Earth in the same amount of de-ionized water.

TS-TDS HRM, 700 mg Total Solids or Total Dissolved Solids/L

1. Dissolve 0.700 g (± 0.002 g) NaCl in deionised water in a clean 1 L volumetric flask containing 500 ml of deionised water.
2. Shake well until all NaCl is dissolved.
3. Make up to 1 L and shake.
4. This standard is prepared quarterly.
5. Label with identification, concentration, analyst, preparation and expiry date of six months and record in Reagent Log QC700, House Reference Material.

Certified Reference Material (peCheck Solids)

peCheck Solids 2; verify and obtain the Consensus value and the Confidence Interval stated on the Certificate of Analysis. (This standard should be added to batches that include samples in the mid-range.)

peCheck Solids 3: verify and obtain the Consensus value and the Confidence Interval stated on the Certificate of Analysis. This standard is added to batches that include Industrial Waste samples and to check high level House Reference Material.

Preparation of the Certified Reference Material (peCheck)

1. To prevent solids sticking to the cap, **Do Not Shake bottle before use.**
2. Put 800 ml of deionised water into a 1-liter volumetric flask.
3. Open the bottle carefully and transfer the contents into the volumetric flask with the use of a funnel.
4. Ensure that the entire standard is added to the flask by carefully rinsing the bottle and the cap three or four times with deionised water.
5. Dilute up to 1000-ml mark with deionised water and mix.
6. Transfer standard solution into a clean and dry polyethylene bottle.
7. Label with the identification, consensus value, and expiry date of six months and record in LIMS. Label the Solution.
8. Make sure to mix for at least 30 seconds before sampling.
9. Run this standard with the first aliquot of every new HRM and use for parameters not covered by the HRM (TDS, TVSS).

Equipment & Supplies

Refer to LIMS Instrument Module and Equipment Logs.

Instruments

- repeater Pipettes, with wide bore tips
- analytical balance, 0.0001g capacity
- analytical balance, 0.01g capacity for sludge analysis
- oven (set at 104°C \pm 1°C)
- oven (set at 180°C \pm 2°C)
- muffle Oven (set at 550°C \pm 50°C)

Ensure that all volumetric dispensers (auto-pipettes) are verified each day in use.

Other Equipment & Supplies

- filter dome apparatus
- desiccators
- gooches; Bitumen Crucibles Coors, 44 mm top diameter, 33 mm bottom diameter, 24 mm height.
- crucibles, H.F. Coors (large)
- crucible Tongs, normal and extra long
- manifolds (stainless steel 3 place vacuum filtration manifolds)
- metal trays (these trays were custom made so that the whole tray is placed inside of the oven(s) and is made to fit in the desiccators as well. This reduces the time required to transfer the items.

NOTE: these trays CAN NOT go inside the 550°C Muffle Oven.

Consumables

- Whatman 934AH glass microfiber filter (or equivalent),
32 mm diameter with 1.5 μ m pore size
47 mm diameter with 1.5 μ m pore size

- Whatman 934AH glass microfiber filters, **Pre-washed & dried filters**, 32 mm diameter with 1.5 um pore size. Delta Scientific, Part No: F92432VOL (Manufactured by Environmental Express, **NO SUBSTITUTIONS!**)
- Plastic Cups (100 mL)
- Disposable Aluminum weighing dish (50 & 150 mL)

Equipment Maintenance

Document equipment maintenance in LIMS equipment log.

Refer to the [Equipment Instruction Manuals^{vi}](#) listed in the References section, page [21](#)

Bar-coded, Pre-weighed Filters

A bar-coded, pre-washed & pre-weighed filters manufactured by Environmental Express, is an approved filter for Total Suspended Solids. The filters are available in the 47 mm size and are ready to use as is. The bar-code contains an identification number and the weight of the filter. Both are also printed in readable font onto a labeled aluminum pan. Opened packages must be kept in the desiccators until ready to use. These filters are currently used at the Water Treatment Plant Laboratory.

Glassware Preparation

Use CLASS A glassware for all volumetric measurements.

- Graduated cylinders, various sizes: **Class A** "to contain"
- Beakers, various sizes

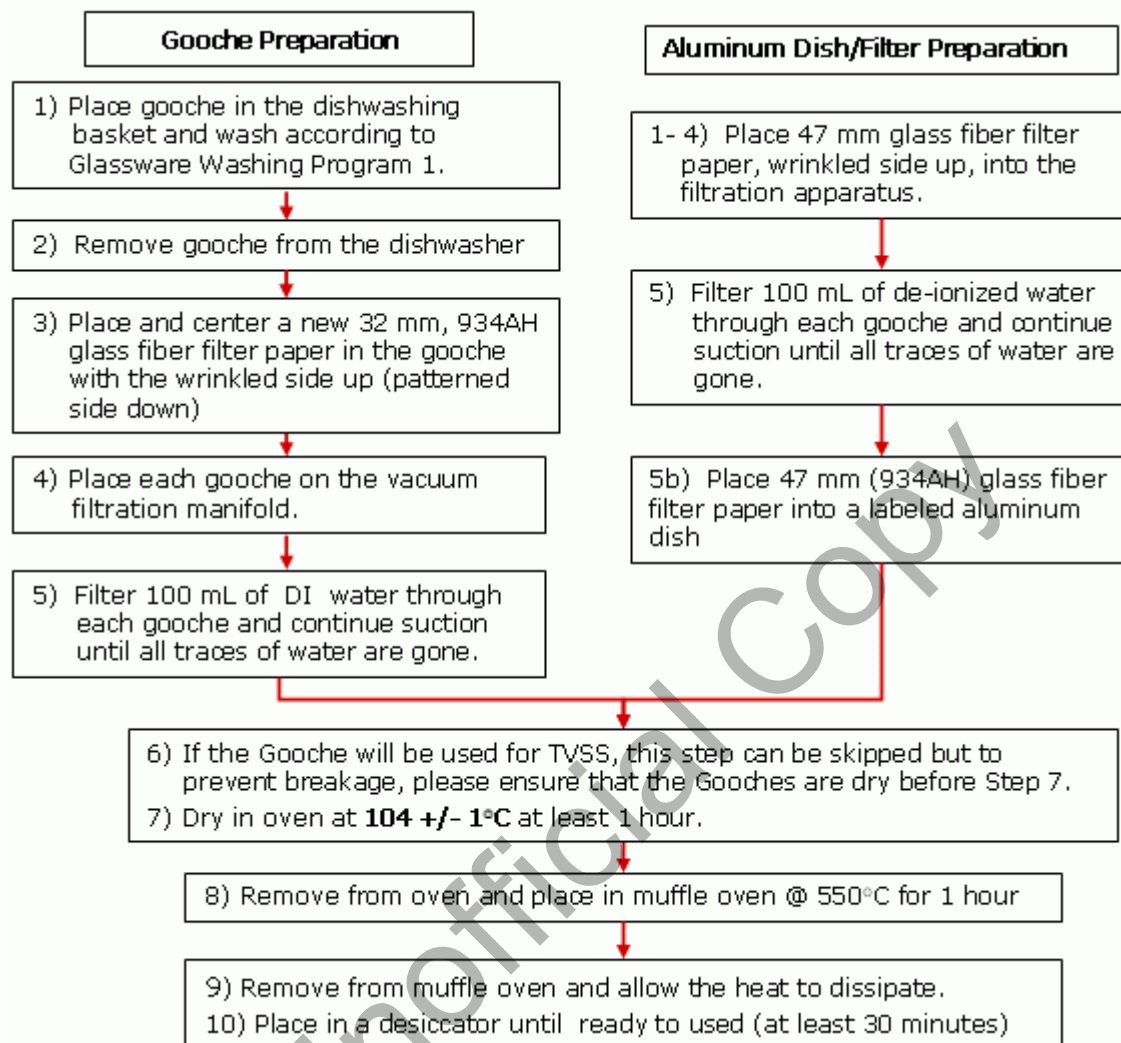
Wash all glassware as described in SOP# 51, Glassware^{vii}, using the Heinicke dishwasher (Program 1), or manually with acid washing and/or soap & water.

Gooche Preparation for Prewashed Filters (Part No:F92432VOL)

A "pre-washed and dried" glass fibre filter paper (purchased from Delta Scientific) may be used for the Total Suspended Solids test and Volatile Suspended Solids. The filter can be placed inside a "prepared" gooche in advance or immediately prior to use and weighing. The gooche only requires washing and drying.

- 1) Place gooche in the dishwashing basket and wash according to Glassware Washing Program 1, which uses a hot cycle and Deionized water in the rinse cycles.
- 2) Remove gooche from the dishwasher.
- 3) **Inspect each gooche for cleanliness and residue. If any residue is discovered, remove the gooche for additional acid washing as described in SOP 51 Glassware washing. (Usually 50% HCl).**
- 4) If the gooches are not dry, then place in 104°C oven for about 15-20 minutes. Remove and cool down.
- 5) Place a F92432VOL filter inside the gooche with the shinier/flatter side down. The gooche requires no further action and can be placed in the desiccator for use.

Gooche/Filter Preparation



Crucible preparation

1. Empty and wipe the inside of the crucible with a paper towel. Acid wash with 50% HCl according to the Manual Acid Washing Procedure in SOP#51 Glassware Washing and observing all of the safety described in the Safe Work Procedures Manual.
2. Wash crucible manually with soap, water and brush until all debris has been cleaned.
3. Rinse with tap water, followed by de-ionized water.
4. Dry crucible in oven at 104°C ±1°C for at least 1 hour.
5. Ignite the clean crucibles in muffle oven for 1 hour at 550°C.

This step is skipped at the Water Treatment Plant lab (and with glass beakers) because no TVSS tests are conducted.

6. Remove from muffle oven, and place onto a heat resistant pad, or tile, and allow the heat to dissipate then place in desiccator until ready to use.

Quality Control Plan

Daily Balance Check

Daily mass balance checks are done and recorded in LIMS.

The balance should be checked by simulating the TSS test as follows; verify with a mid-range weight (20 g), tare the balance with the weight and verify again by adding a low range weight (500 mg or less) to the balance. Remove the weights, re-zero (tare) and verify with a high range (similar to a crucible with sample).

The daily balance check for the top-loading balance is initially documented on hardcopy and entered in the LIMS. The worksheet tab "Balance Cal Log" is in "SolidsForms.xls".

Auto-pipette verifications are done daily and recorded in LIMS.

The 'Internal Calibration' function on Balance3 is performed at least once per week and recorded in the LIMS.

Batch Creation

All sludge(s) & cakes are weighed to two decimal places using a top-loading balance and documented in the corresponding daily workbooks. All workbooks are bound and created by ASB staff using the approved templates and cover pages, listed in Appendices, [Tab 3: Worksheets](#), page 20.

Unless otherwise indicated, all charts are available from the following spreadsheet [ChartGenerator.xlsx](#);

Method Quality Control

Include and run the blank and CRM or HRM (or combination of) at the beginning of the batch and after every 25 to 30 samples. If a batch contains samples with expected results in the low and medium/high range, consider batching "similar" samples and using two different levels of HRM/CRM.

- Filter 200mL of deionised water into a gooch and use it as a Blank with every batch of samples run.
- Analyze 25 to 50ml of the TSS HRM standard with every batch of TSS.
- Analyze 25 to 50ml of CRM for TSS with every new bottle of HRM.
- Run one TDS HRM or CRM with every batch of TDS.
- Run one TS HRM or CRM with every batch of Total Solids
- Analyze one replicate or duplicate sample for all test parameters in the batch (TSS, TVSS, TS, TVS and TDS) with every batch of plant samples. Preferably, separate large batches into two, one with all the volumetric measurement (weight/volume) and one with the gravimetric measurement only (weigh/weight), TS%, TVS%.

When two separate samples are collected at the source, then these shall be documented as duplicates. This is feasible with any grab sample.)

- Reweigh another sample to confirm the completeness of the drying and desiccating and balance stability, (Dry the sample for an additional 30 minutes and desiccate for another 30 minutes then reweigh).
- Record the values for the QC samples in the LIMS QC database.
- Compare the results with the control limits.

- If any quality standard or blank exceeds the upper or lower control limits, repeat the analysis of the corresponding quality standard or blank.
- If the value exceeds the upper or lower control limits a second time, generate a non-conformance report and carry out corrective actions. This may include cleaning of glassware, preparation of new standards, and/or repeat analysis of the batch.

Acceptance Criteria

	TSS & TVSS (mg/L)	TDS mg/L	TS mg/L	%TS (sludge/ww)	%TVS
Sample Blank	±3 mg/L	±3 mg/L	±3 mg/L		
HRM/CRM*	±11%	±9.3%	±11%	±11%	±11%
Duplicate	±11%	±10%	±7%	±7%	±4%
Repeat (reweigh)	±6%		±6%	±7%	

*The daily use standard (CRM or HRM) recovery values shall be plotted on Control Charts and shall be within the established upper and lower control limits. These values shall also be entered into the QC table in the LIMS. Where the CRM is supplied with a value and confidence limits, then those shall be followed unless otherwise stated.

Non-Conformance and Corrective Action

If the CRM or HRM is outside the control limits for any test, then reject the batch (about 25 samples per CRM) and re-analyze. If the CRM is over the limits, re-dry, re-weigh and recalculate the results. If the control limits are still exceeded, then reject the batch and re-analyze.

If any of calibration verification, duplicate recovery, replicate recovery spike recovery or control standard value is outside of the control limits then

- Document the non-conformance on the Control Chart.
- Carry out corrective action to resolve the problem. (See Quality Manual for corrective action procedure.)

Document the corrective action and maintain the records for audit and review.

Proficiency Testing

The Analytical Services Branch is accredited to ISO 17025: 20005 General Requirements for the Competence of Testing and Calibration Laboratories and actively pursues proficiency for Solids at both laboratory locations. Participation in the CALA Proficiency Testing for Solids, began in 2000 at 2230 Main St. and in 2011 at the Water Treatment Plant. TSS, TDS and TVSS are included in the C-04A Test Group Solids. TVSS or TVS are not analyzed at the Water Treatment Plant because the test is not required.

The laboratory also participates in the Phenova Proficiency Testing program two to four times per year for TS and TVS at 2230 Main St. and for TS at the Water Treatment Plant Laboratory. Additional parameters are done if included and part of the sample or group.

Test Procedure

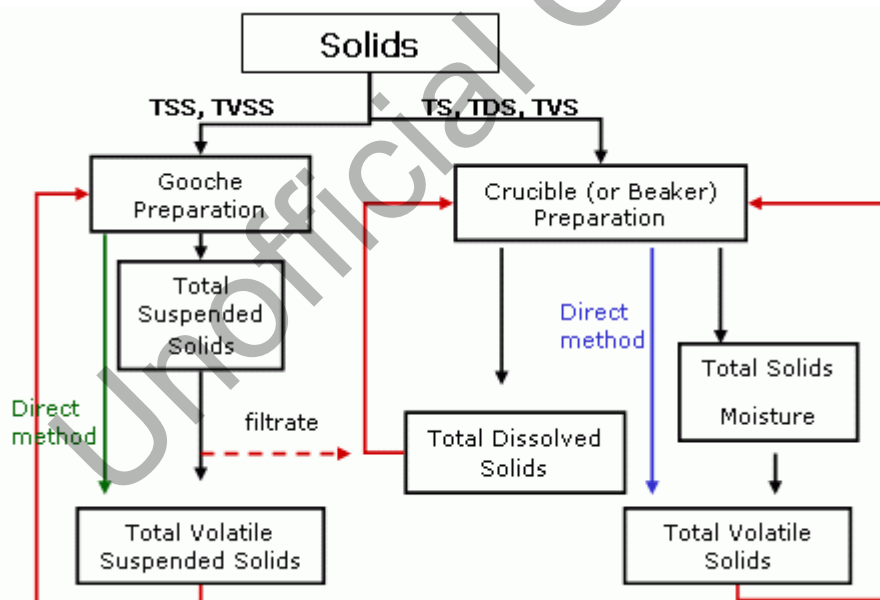
Taking Aliquots (sub-samples)

For larger volumes, place the sample, up to 1 litre into a large beaker so there is sufficient room for mixing. Using a small ladle, stir the contents until a vortex is obtained. Take several small ladle full and transfer into a graduate cylinder. Always use the smallest graduate cylinder that can hold the total volume. Use **CLASS A** "TC" (to contain) glassware.

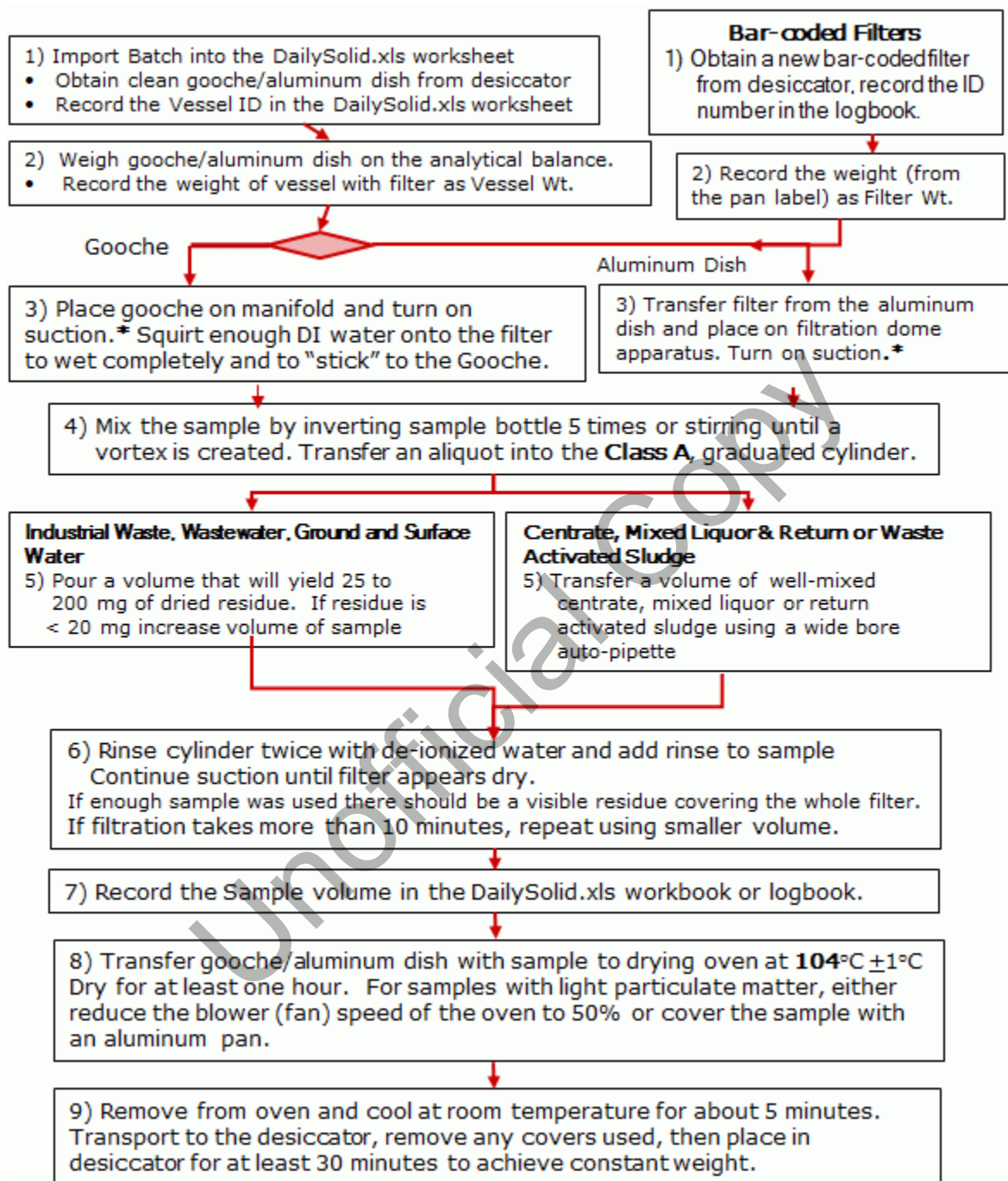
- 1-10 mL, use an auto-pipette
- 10-20 mL, use a 10 mL auto-pipette, two times
- 20-25 mL, use a 25 mL graduate cylinder
- 25-50, use a 50 mL graduate cylinder
- 50-100, use a 100 mL graduate cylinder
- 100-500, use a 250 mL graduate cylinder (twice if needed)

For smaller volumes, create a vortex by manually stirring the sample or by using a magnetic stirring bar and stirrer. Using a wide-bore pipette tip, take the aliquot midway into the sample (depth) and halfway between the wall of the beaker and middle of the vortex. Transfer directly to the filter/media.

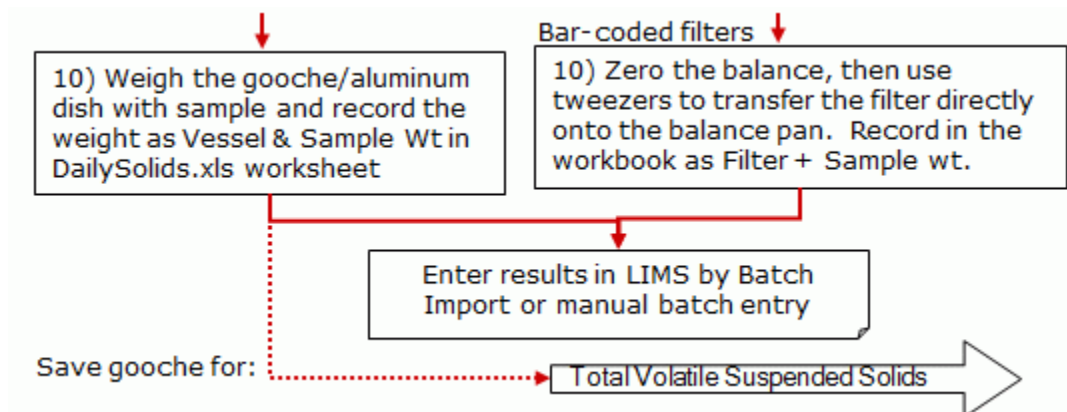
Solids Methods



Total Suspended Solids

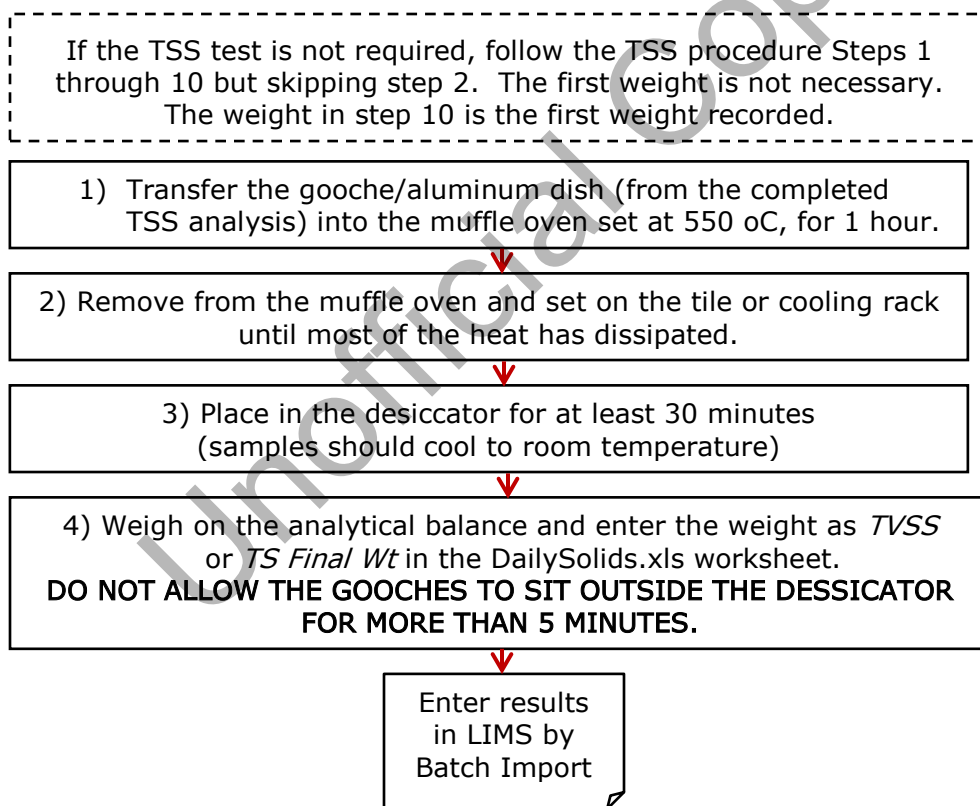


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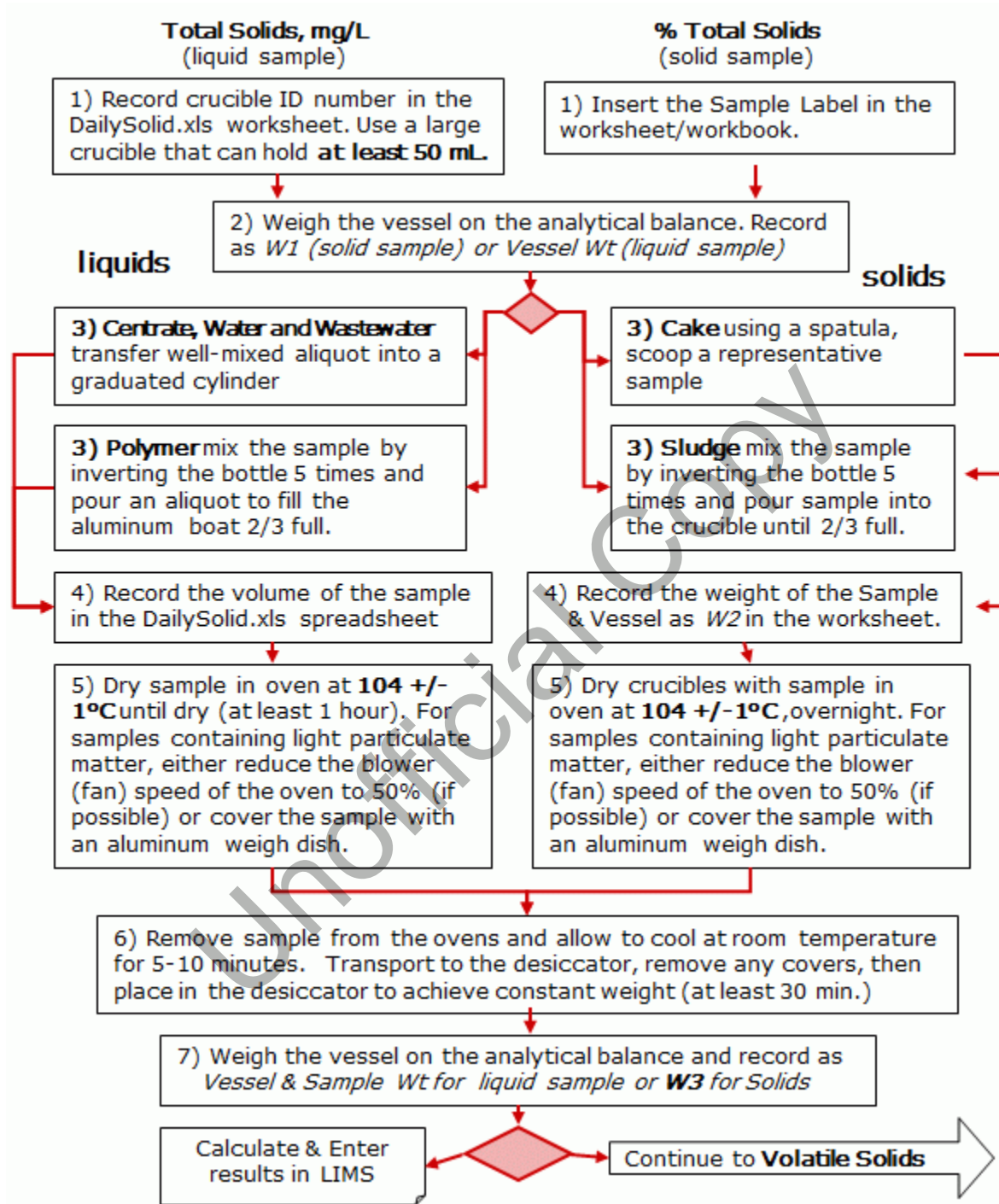


*Note 1: Rinse the plastic manifold (vacuum dome assembly) with 10% HCL and with copious amounts of DI water between every sample.

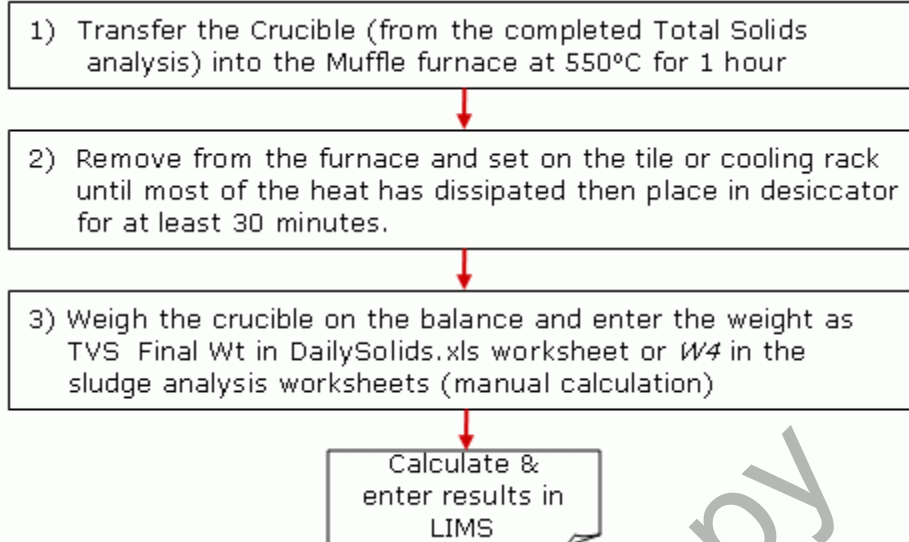
Total Volatile Suspended Solids



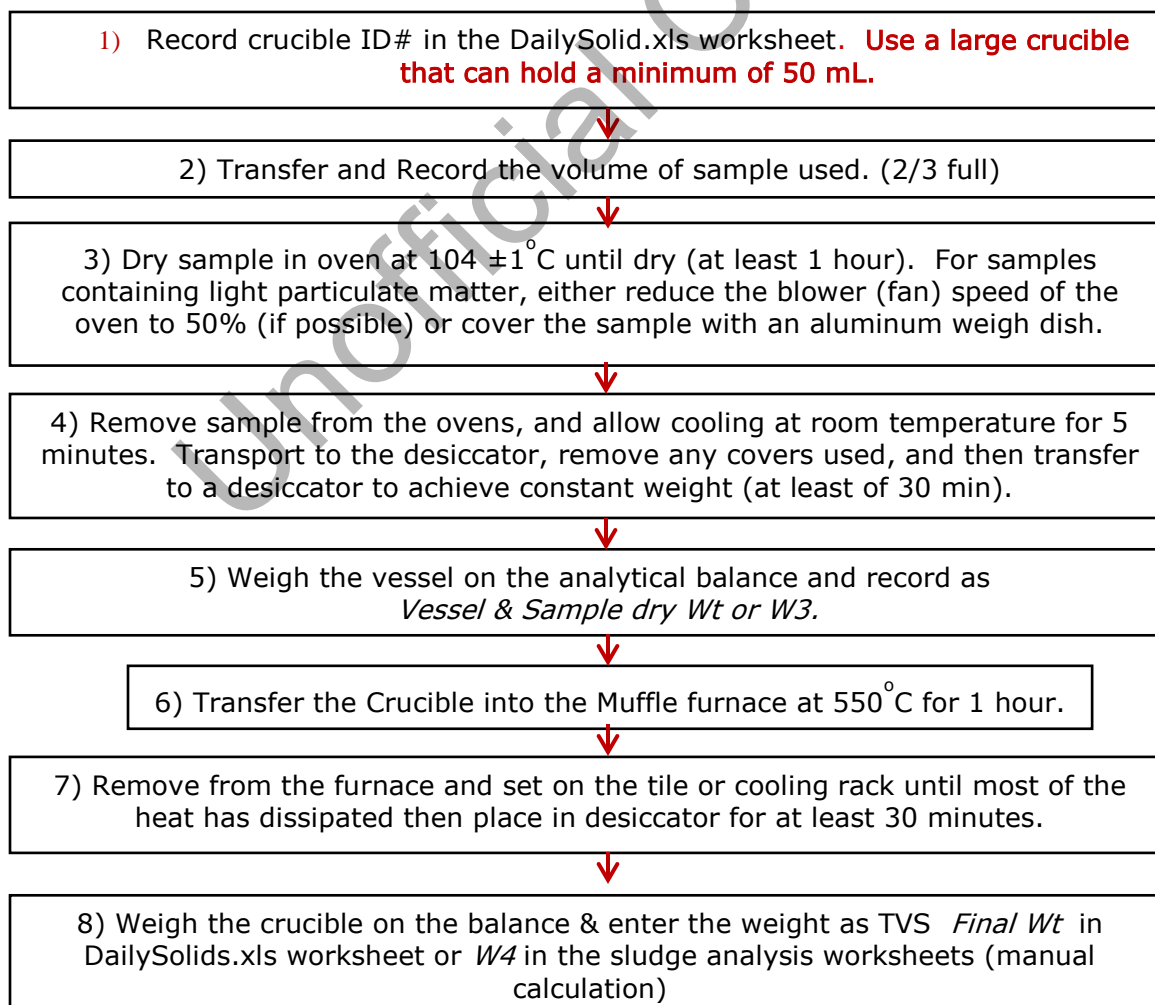
Total Solids



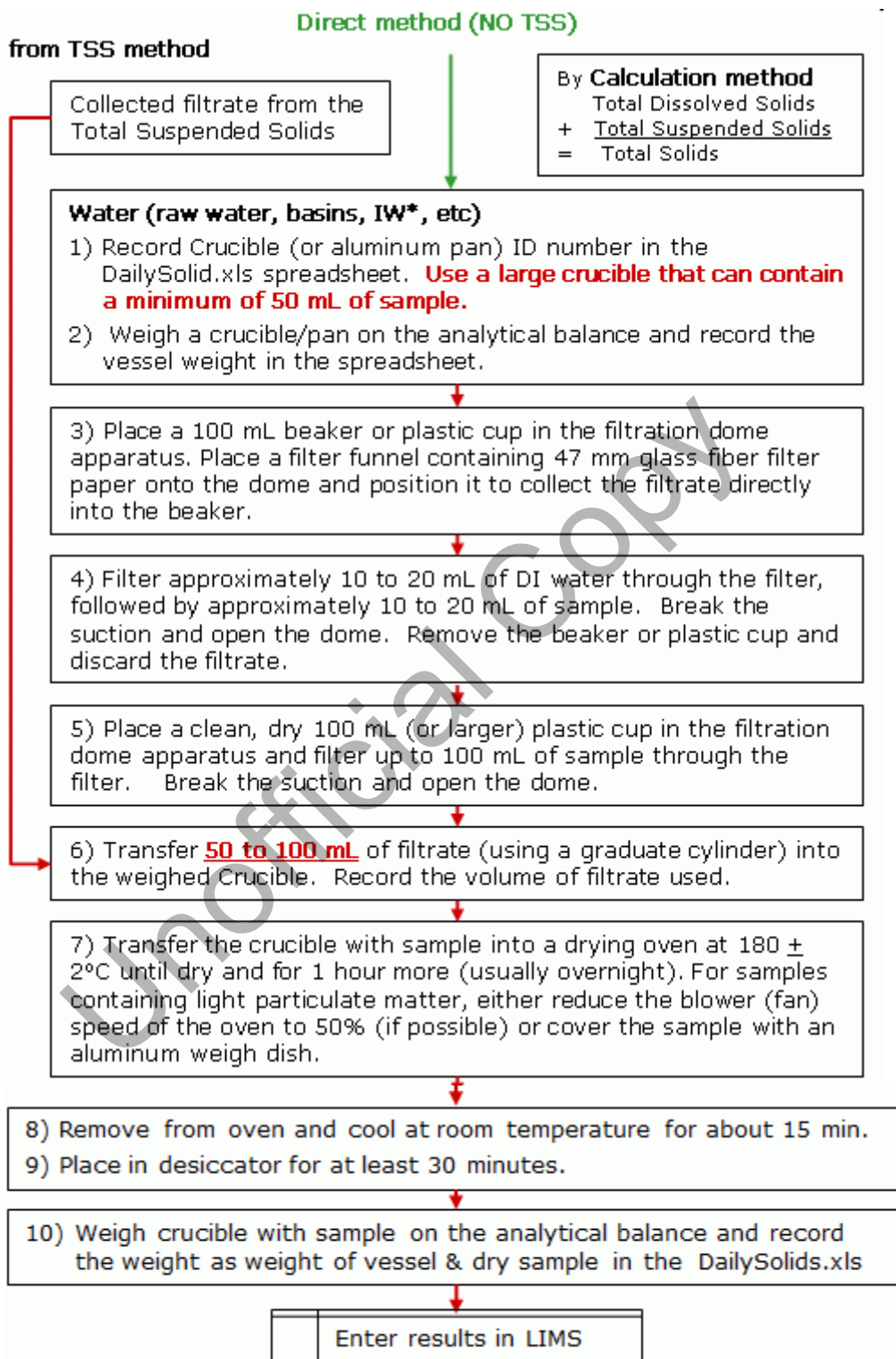
Volatile Solids (following the TS)



Volatile Solids, direct method for liquids



Total Dissolved Solids



*Note 2: Some Industrial Waste samples and samples with very high solids/particulate content tend to clog the filter immediately. These samples are centrifuged at 4000 rpm for 20 minutes prior to filtration. A note is entered in the test comment or job comment indicating that the sample was centrifuged.

Data Analysis & Calculations

Calculations are automatically done when using the DailySolids.xls program. A manual calculation to verify the formula must be done and recorded at least once after any revisions or changes are incorporated into the DailySolids.xls program.

Manual Calculations

TOTAL SOLIDS (%TS)		TOTAL VOLATILE SOLIDS (%TVS)
WET WEIGHT (g)	DRY WEIGHT (g)	
W2 - W1	W3 - W1	W3 - W4
W5 g	W6 g	W7 g
	%TS	%TVS

W1 = weight (g) of vessel (gooche crucible, crucible, aluminum dish, or beaker)

For total suspended solids **W1** = weight of prepared gooche + filter

W2 = weight (g) of the vessel & sample.

For total suspended solids **W2** = weight of dried gooche + filter + sample

W3 = weight of vessel and dried residue after drying at 104 ±1°C or 180 ±2°C

W4 = weight (g) of residue after ignition at 550 °C.

W5 = W2-W1 or sample weight

W6 = W3 - W1 or dry sample weight

W7 = W3 - W4 or weight of volatile component

V = sample volume (mL)

$$\text{Total Suspended Solids mg/L} = \frac{(W2 - W1) \times 1000 \times 1000}{V \text{ (mL)}}$$

$$\text{Total Volatile Suspended Solids mg/L} = \frac{(W3 - W4) \times 1000 \times 1000}{V \text{ (mL)}}$$

Or (W7x 1000 x 1000/V (mL))

$$\text{Total Solids mg/L} = \frac{(W2 - W1) \times 1000 \times 1000}{V \text{ (mL)}} \quad \text{or } (W5 \times 1000 \times 1000/V)$$

$$\% \text{ Total Solids} = \frac{(W3 - W1) \times 100}{(W2 - W1)} \quad \text{or } (W6 \times 100/W5)$$

$$\% \text{ Total Volatile Solids} = \frac{(W3 - W4) \times 100}{(W3 - W1)} \quad \text{or } (W7 \times 100/W6)$$

$$\text{Volatile Solids (mg/L)} = \frac{(W3 - W4) \times 1000 \times 1000}{V \text{ (mL)}}$$

$$\text{Total Dissolved Solids mg/L} = \frac{(W2 - W1) \times 1000 \times 1000}{V \text{ (mL)}}$$

Or by calculation only:

Total Dissolved Solids mg/L = Total Solids mg/L – Total Suspended Solids mg/L

% Moisture = 100 - %Total Solids

All samples recorded in the DailySolids.xls spreadsheet are automatically calculated using the "Get Formula" function. The Quality Control is also calculated using the "QC Calculator" function.

Safety

Lab coat and safety glasses/goggles must be worn in the laboratory at all times.

Sewage samples contain pathogenic organisms. Eliminate contact above the chest, i.e. protect all exposed skin facing the material.

Read all MSDS caution labels.

Refer to each chemical MSDS for detailed hazards, available on the City of Winnipeg's intranet site, "Wellness" page^{ix}.

Refer to Environmental Standards Division, Health and Safety Manual^x, and applicable Safe Work Procedures in the Safe Work Procedures Manual^{xi}.

See Work Safe Procedure Manual for the following;

OVEN USE

CHEMICAL PREPARATION AND HANDLING

ACID WASHING

Revision History

Previous Revisions are maintained in the obsolete version 4.3.

Version 5.0	November 1, 2013	Reviewers: H.Demchenko, G. Levesque
<u>Scope</u> , Range, MDL, Bias, Precision, and Uncertainty recalculated		p. 3
<u>Fit for Use Statement</u> , Rewritten for clarification		p. 3
<u>Sample Requirements</u> , updated Sampling #17 reference.....		p. 4

Current Version 5.1

Version 5.1	October 10, 2014	Reviewers: J. Jones, G. Levesque
Removed most hyperlinks to external documents/folders.		
<u>Introduction and Scope</u> , referenced method		p. 3
<u>Sample Requirements</u> , analyze within 2 hours if not refrigerated.....		p. 4
<u>Glassware Preparation</u> , may be washed manually		p. 8
Inspect & acid wash gooches that are still dirty after going through the dishwasher		p. 8
<u>Crucible (beaker) Preparation</u> , Crucibles are not put in the dishwasher (step removed)		p. 9
<u>Quality Control Plan</u> , Internal balance calibration is performed on Balance3.....		p. 10
<u>Appendices</u> , added Tab 6: Daily Balance Calibration Check.....		p. 20
<u>References</u> , added balance user manual references		p. 21
Endnotes, new section in References for listing file and folder locations in one place		p. 21
Update flow charts on pages 13 to 17		

Next Review; No Later than **October 20, 2015**

Sign the following only if there are no significant changes required at the next review.

Reviewed By: _____ Review Date: _____

Document Control

After each calendar year, the hard copy charts and logs are turned into electronic format, as a single PDF file. These are filed in the Methods\Solids\QC RECORDS (Historical PDF) folder and hard copies are destroyed in accordance with the documented policy in the Quality Manual.

All workbooks (with manual calculations) are catalogued, filed and maintained for a period of 7 years. (Mark the destruction date as the workbook year + 8).

A copy of all electronic worksheets ([created by DailySolids.xlsx](#)) and imported into the LIMS is maintained in the generic analyst (wwdanalyst) folder; [G:\Solids\](#)

Certificates of Analysis are also converted in PDF formats and filed in the Certificate of Analysis folder, and simply named according to the LIMS Chemical ID#.

Appendices

Tab 1: Control Charts

House Reference Standard
Certified Reference Material

Tab 2: Reagent Preparation Logs & Certificates

Certificates of Analysis for CRM (originals scanned & filed in Certificates of Analysis folder)

[Reagent Log: TSS-HRM1000](#)

Reagent Log: TSS-HRM100

Reagent Log: TS QC700

Tab 3: Worksheets

Word Document templates for Manual Data Calculations

- NE Dewatered Sludge Worksheet ([WkshtDewateredSludge.doc](#))
- NE & SE Raw Sludge Worksheet ([WkshtNESERawSludge.doc](#))
- WE (Fermentor) Sludge Worksheet ([WkshtFermentorSludge.doc](#))

Tab 4: DailySolids.xls Spreadsheets (Instruction Manual)

Tab 5: Method Validation

Tab 6: Daily Balance Calibration Checks

References

Supporting Test Methods

SOP#51, Glassware Washing
SOP#52, Mass and Volume Calibration
SOP#50, LIMS User Manual
SOP#49, Temperature

Equipment Instruction Manuals

Electronic Copies (PDF documents)

Ovens

Operations Manuals and Parts List, Series 1249 (Thermolyne Oven/Furnace)
Temperature Adjustment Procedure, Fisher Isotemp Ovens
Operating Manual, APT.line™ FED (E2) (Binder Oven)

Balances

[MettlerAE200.pdf](#)
[OhausDiscoveryBalance.pdf](#)
[Sartorius A200S_Manual.pdf](#) (Water Treatment Plant)

Hard Copies

Balance Manuals located in balance room [at 2230 Main Street](#) (magazine rack).

A hard copy of the "Binder FED(E2)" Oven11 is clipped onto the oven.

Computer Software Documentation

[WinWedge Help File](#) on the "Balance Computer"

Available through the LIMS, Methods,

- [DailySolids.xls](#) Spreadsheet, Instruction Manual (copy in method binder)
- [PipetteVer.xls](#) Instruction Manual (Excel program)

Endnotes (file and folder locations)

ⁱ Standard Methods for the Examination of Water and Waste Water, 2504A, 2540B, 2540C, 2540D, 2540E, 2540G, Approved 1997, Editorial Revisions 2011.

[N:\Environmental Standards\Analytical Services\Standard Methods](#)

ⁱⁱ Method Validation Data is in [N:\Environmental Standards\Methods\Solids\MethodValidation](#)

ⁱⁱⁱ SOP#17 Sampling, and Supporting Work Instructions, SWI #17 in [N:\Environmental Standards\Methods\Sampling \(SOP 17, SWI 17\)](#)

^{iv} SolidsForms.xlsx, in [N:\Environmental Standards\Methods\Solids \(SOP 12\)\FORMS](#)

^v Certificates of Analysis (COA) are in

[N:\Environmental Standards\Analytical Services\Certificate Of Analysis\(CRM,Stds\)](#)

^{vi} Equipment Manuals, where available, have been created and copied into the following folders;

[N:\Environmental Standards\Analytical Services\Instrument & Software\Ovens & Furnaces](#)

[N:\Environmental Standards\Analytical Services\Instrument & Software Manuals\Balances](#)

^{vii} SOP#51, Glassware in [N:\Environmental Standards\Analytical Services\SOP \(PDFs\)](#)

^{viii} Chart Generator in [N:\Environmental Standards\Analytical Services\Charts](#)

^{ix} <http://citynet/intrahom/toc/wellness.stm>

^x SafetyManual, (word document) in [N:\Environmental Standards\Safety\SafetyManual](#)

^{xi} Safe Work Procedures (individual PDF) located in

[N:\Environmental Standards\Safety\Safe Work Procedures\Safe Work Procedures pdf](#)